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Description

Herbicidal compositions comprising post-emergence herbicides for soil application

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The invention relates to the technical field of herbicides, and in particular of herbicides for soil application.

For controlling undesirable harmful plants, the user can choose from a large number of herbicides which can be applied depending on the biological properties of the herbicides, the type of harmful plants to be controlled and the type of useful plants. Here, it has also to be taken into account that numerous herbicides, owing to their intrinsic mechanism of action, can be used either exclusively by the pre-emergence method or exclusively by the post-emergence method. Both methods and the herbicides used for these methods have certain advantages and disadvantages. Disadvantages of the post-emergence method where, for example, herbicides with foliar action such as bilanafos, diquat, glufosinate-ammonium, glyphosate and paraquat are also used, are considered to be, for example:

- damage to useful plants by overdosage
- depending on the type of herbicide used, on the harmful plants to be controlled and/or the useful plants in question, a need to repeat the application several times.

The last mentioned disadvantage is particularly grave, especially from the point of view of time-economic farming.

On the other hand, numerous herbicides which can be used by the post-emergence method have considerable advantages, in particular also from an ecological point of view, since in many cases they have more favorable toxicological and ecotoxicological properties than the herbicides which can be used by the pre-emergence method. Depending on the particular application situation, in many cases it would therefore be desirable for the user to be able to use a post-emergence herbicide under the conditions of a pre-emergence application, i.e. prior to the emergence of the plants or, if appropriate, simultaneously with the sowing of the useful plants. However, hitherto such an application has, in particular in the case of herbicides with foliar action, not been possible, for reasons of the mechanism of action on

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which these herbicides are based, since herbicides with foliar action in the form of the herbicidal compositions of the prior art, for example, are only taken up via the green parts of the plant, for reasons of their leaching properties or else for reasons of their degradation behavior in the soil.

5 Glufosinate-ammonium (2-amino-4-(hydroxymethylphosphinyl)butanoic acid), for example, which can be used as herbicide with foliar action is known to be decomposed rapidly in the soil, so that it is not capable of displaying any herbicidal action (G. Hoerlein in "Reviews of Environmental Contamination and Toxicology", vol. 138, Springer-Verlag; "The Pesticide Manual", 11th Edition, 1997, British Crop Protection Council). The herbicide 10 glyphosate (N-(phosphonomethyl)glycine), which likewise has foliar action, is known to be adsorbed strongly by the soil and to be degraded therein, so that it is not available to the plant in sufficient amounts (L. Torstensson in "The Herbicide Glyphosate", Butterworths, pp. 137-150).

15 Hereinbelow, the term "herbicide" is, depending on the context, to be understood as meaning the pure herbicidally active compound or the herbicidally active compound in formulated form, i.e. the herbicidal composition. The terms "pre-emergence" and "post-emergence" are here to 20 be understood as referring to the time of the emergence of the harmful plants, i.e. a treatment by the pre-emergence method means a treatment with a herbicide prior to the emergence of the harmful plants and thus prior to the formation of green parts of plants. Analogously, a treatment by the post-emergence method means a treatment with a herbicide after 25 emergence of the harmful plants.

In the field of crop protection and, accordingly, also in the field of the control of harmful plants, there have been repeated attempts to enhance the use properties of a given active compound, such as, for example, its 30 activity spectrum, its persistence or the required application rate, for example by modified formulations or application methods. Thus, Weed Research, 1997, 37, 19-26 examines whether the activity of the herbicide chlorsulfuron can be enhanced and its tendency to leach can be reduced by using a controlled-release formulation. US 5,674,519 discloses that the 35 tendency of certain crop protection agents including herbicides to leach in the soil can be reduced by a formulation in which the active compounds are present in microencapsulated form. WO 98/05205, too, describes a particular form of encapsulating crop protection agents which leads to an increased activity and a reduced susceptibility to leaching by rain.

Furthermore, WO 99/26474 and WO 99/26743 describe methods for releasing active compounds using cyclodextrins or carbohydrates.

However, none of the publications mentioned indicates that it may be
5 possible to use post-emergence herbicides pre-emergence by employing suitable measures.

Accordingly, it is an object of the present invention to make possible the use of post-emergence herbicides by the pre-emergence method. This
10 object is achieved by herbicidal compositions, comprising an effective amount of one or more post-emergence herbicides and an amount of a carrier material from the group consisting of fullers earth, aerogels, high-molecular-weight polyglycols and polymers based on acrylic acid, methacrylic acid and copolymers thereof.

15 Here, the compositions according to the invention should only comprise post-emergence herbicides which, in the form of the herbicidal compositions of the prior art, can be used exclusively by the post-emergence method, i.e. after emergence of the undesirable harmful plants.

20 Surprisingly, post-emergence herbicides which are present bound to the carrier materials mentioned above display herbicidal action against undesirable harmful plants when used by the pre-emergence method, i.e. when the herbicidal compositions have been applied prior to the
25 emergence of the harmful plants.

Usually, the compositions according to the invention comprise

- a) from 0.001 to 48% by weight of one or more post-emergence herbicides,
- 30 b) from 2 to 90% by weight of a carrier material and
- c) from 0 to 97% by weight of a solvent.

Particularly suitable post-emergence herbicides are those from the group of the herbicides with foliar action. Preference is given to herbicides from the
35 group consisting of bilanafos, diquat, glufosinate-ammonium, glyphosate and paraquat. Particular preference is given to glufosinate-ammonium. The abovementioned herbicides are known, for example, from "The Pesticide Manual", 11th Edition, 1997, British Crop Protection Council. The herbicides in question can, of course, also be employed in the commercial

salt form, as known, for example, from "The Pesticide Manual".

It is a further advantage of the compositions according to the invention that they are present in solid form and can be used, for example, in the form of granules. The user can apply this solid presentation form directly to the area to be treated, i.e. without it being necessary to prepare a spray liquor. To this end, they are, depending on the type of useful plants and the type of expected harmful plants to be controlled, applied onto the soil to be treated, worked into the soil or applied by side application.

Side application means that the herbicide is applied in the soil below the seeds. Here, it is particularly advantageous that the application of the herbicidal compositions can also take place in the same operation as the sowing of the crop plants, resulting in the soil being subjected to reduced mechanical stress.

The herbicides which can be used for the compositions according to the invention can be processed to the compositions according to the invention in the form of the pure active compound or else in the customary commercial formulation together with carrier materials and, if appropriate, additional substances, in the manner described further below. A particular advantage of the compositions according to the invention is their good environmental compatibility. Owing to the fact that the herbicides, in contrast to conventional application methods, are not sprayed in liquid form but applied in solid form, there is no risk of uncontrolled drift of spray mists onto the user and adjacent areas and plants. Moreover, environmentally compatible formulation auxiliaries are used as they are either - like the high-molecular-weight polyglycols and polymers of acrylic acid, methacrylic acid and copolymers thereof - biodegradable to unobjectionable substances or - like fuller's earth - show environmentally neutral behavior.

The herbicidal compositions are prepared by mixing the active compounds, carrier materials and, if appropriate, further additives in question according to methods known to the person skilled in the art. Granules, for example, can be prepared by the methods described in EP-A - 0 413 267.

Depending on the carrier material, the substances in question can be present in solid or liquid or dissolved form. In the preparation of granules, it is advantageous for the active compounds and additives to be incorporated to be present in liquid or dissolved form. Here, these substances can be applied uniformly in the known manner for example by spraying, pouring,

dropwise addition, treatment in a fluidized bed, concrete mixer, tumble mixer, etc. It is also possible to oversaturate these carrier materials with solutions of the active compounds and additives in question to form, for example, gels, or to remove excess water by drying. In the case of the preparation of melt granules, for example melt granules based on polyethylene glycol, active compounds and additives are preferably incorporated in solid form and extruded. Also possible is tabletting, pelletizing, the preparation of flakes and comminution to powders by breaking or grinding. In all preparation processes, aftertreatment by comminution may take place. It is also possible to compact very fine granules using suitable adhesives to avoid the formation of dust. It is furthermore possible, if appropriate, to add additives to improve, for example, the flowability of the granules or their wettability.

Depending on the intended purpose, the herbicidal compositions may additionally comprise other substances which can be used in crop protection, such as pre-emergence herbicides, plant growth regulators, fungicides, insecticides, safeners ((herbicidal) antidotes), nutrients, preservatives, seed dressings and fertilizers. The selection of the pre-emergence herbicides, plant growth regulators, fungicides, insecticides and seed dressings to be added, if appropriate, depends on the type of useful plants and the type of expected harmful plants, fungi, insects and feed pests to be controlled. In principle, it is possible to use all commercial substances which are usually employed in crop protection. Suitable nutrients and fertilizers are, in particular, aqueous ammonium nitrate/urea solutions and NPK solutions such as 12-6-8, 8-8-6, 5-8-10, and also ammonium sulfate and/or ammonium nitrate solutions.

Preferred carrier materials are aerogels, polymers based on acrylic acid, methacrylic acid and copolymers thereof and also high-molecular-weight polyglycols.

When using the compositions according to the invention, it has to be ensured that the useful plant is sufficiently tolerant to the herbicide employed. In addition to useful plants in which sufficient tolerance to herbicides has been achieved by targeted genetic manipulation, this includes plants which, such as, for example, barley, have sufficient tolerance to some herbicides even without genetic manipulations. The compositions according to the invention can be used particularly

advantageously in crops of useful plants from the group consisting of cereals, corn, soyabean and rapeseed. They are preferably used in crops which are sufficiently tolerant to the herbicide in question owing to genetic manipulation.

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As in the case of other herbicidal compositions, it is, of course, in the case of the compositions according to the invention also possible and in some cases advantageous to provide the herbicide with additives such as surfactants, wetting agents, emulsifiers, adjuvants, ammonium salts, preservatives, colorants, antifoams, tackifiers, solvents, buffer systems and UV stabilizers. Depending on the type and purpose of the additives, these can be processed together with the herbicide in a formulation or else be present and subsequently used separately from the herbicide. These additives serve to improve application properties. The use of such additives may be advantageous to enhance, for example, the persistence of the herbicide. Preservatives are used, for example, to slow down the biological degradation of active compounds and/or formulation auxiliaries.

Suitable surfactants are, for example, condensates of formaldehyde and phenol and/or naphthol, in each case with or without sodium bisulfite, such as Tamol NNO from BASF, Rapidamin-Reserve C from Clariant or Galoryl MT800 or DT201 from CFPI; C₁₂-C₂₄ fatty alcohols having 2 to 40 EO and/or PO, if appropriate phosphated and/or neutralized with alkanolamine, alkali metal or ammonia; di- and tristyrylphenyl analogs of the fatty alcohols mentioned above, such as, for example, the Genapol series from Clariant, Grafol types from Henkel and Soprophor types from Rhodia; alkyl ether sulfates such as Genapol LRO® from Clariant; alkyl- and/or alkenylsulfonates such as Hostapur OS from Clariant, ligninsulfonates such as Ufoxane 3A and Vanisperse CB from Booregard; Reax types from Westvaco; N-methyltaurides such as Hostapon T from Clariant; sulfosuccinic monoester salt such as Hoe S 1728 from Clariant; alkyl polysaccharides such as Plantaren APG 600 from Henkel; ethoxylated C₁₂-C₂₄-fatty amines, such as the Genamin types from Clariant. Suitable solvents are, in each case depending on the intended purpose, alcohols, diols, polyols, N-substituted pyrrolidones, ketones, aldehydes, ethers, polyethers, paraffins, aromatic compounds, heteroaromatic compounds, cycloalkanones, dimethylsulfoxide, tetrahydrofuran and water. It is advantageous to employ water.

Suitable preservatives are, for example, Bronidox L from Henkel, Mergal types from Riedel de Haen, Proxel from ICI, ascorbic acid derivatives, benzoic acid derivatives, formaldehyde, citric acid, preservatives from the Kathon series from Rohm & Haas and brohopol from BASF.

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Suitable adjuvants are, for example, alkyl polysaccharides or lauryl ether sulfates.

10 Suitable carrier materials from the group of the high-molecular-weight polyglycols are, in particular, polyethylene glycols having a molecular weight of from 2000 to 40,000 (PEG 2000 to PEG 40,000). Suitable carrier materials from the group of the aerogels are, in particular, the aerogels described in EP-A 0 171 722.

15 It is particularly advantageous to add a substance such as ammonium sulfate, ammonium nitrate and one of the surfactants mentioned above, in particular a surfactant from the group of the alkyl ether sulfates.

20 The use of the herbicidal compositions by the pre-emergence method is novel and also forms part of the subject matter of the invention.

Using the herbicidal compositions according to the invention, it is possible to control undesirable harmful plants by the pre-emergence method. This method is new and also forms part of the subject matter of the invention.

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The invention is illustrated by the embodiments that follow.

A. Formulation examples

30 By way of example, the qualitative and quantitative (in % by weight) composition of numerous herbicidal compositions according to the invention is listed in Tables 1 to 4.

Table 1
Herbicide (active compound)

No.	Herbicide (active compound)	Carrier materials	Additives
1	Basista 50% (glu-tosinate)	Ludusorb AF1	Aerogel C373
2	Basista 150 g/l (glu-tosinate)	Ludusorb AF2	Water
3		Aerogel P88	
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Basista 150 g/l: 13.5% of glufosinate-ammonium, 58.81% of Genapol LRO, 10.0% of Dowanol PM, 0.25% of Fluowett PL80, 0.005% of Duasyn acid blue AE, water ad 100%

Table 2

Herbicide no.	Carrier materials	Additives	Water
23	2	40	58.00
24	2	35	63.00
25	2	30	68.00
26	85 15		
27	95 5		
28	2	81	5.99
29	2	81	5.99
30	2	86	5.99
31	2	86	1.99
32	2	40	42.95
33	2	40	39.25
34	85	5	10
35	85	5	
36	85	5	
37	11	84	
38	80	5	15.00
39	80	5	9.00
40	2	75	6
41	2	75	17.00
42	2	75	14.00
43	2	75	17.00
44	1	4	14.00
45	1	1	99
			98

Table 3

Table 4

Herbicide	Carrier mat.	Additives	Water
	68	Aerogel P88	38.00
	69	④Basta 99%	38.00
	70	④Basta 50%	38.00
	71	no.	38.00
	72	glycol 20,000	38.00
	73	Hostapon T	38.00
	74	Geropon SDS	38.00
	75	Dispersant SI	38.00
	76	Galoryl MT	38.00
	77	ECD 1736	38.00
	78	Hostaputr SAS30	38.00
	79	KPE Phospholan	38.00
	80	Genapol O	38.00
	81	Mowiol 18-88	38.00
	82	Tylose H2O	38.00
	83	Beroil 992	38.00
	84	Soprophor 3 D33	38.00
	85	Soprophor F1	38.00
	86	Emulsogen 1860	38.00
	87	Rhodonat SGE	38.00
	88	Supragill WP	38.00
	89	Sipemata 22 S	38.00
	90	Mergali K9N	38.00
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Table 1a - Combinations

no.	Basista 50%	Basista 99%	Herbicide	Carrier material	Additives									
					Aerogel P88	polyethylene glycol 20,000	Hostapur OSB	propylene glycol	ammonium nitrate	Bronidox L	diasyn acid	blue AE	Stepanol ME dry	Mergal K9N
87	2.0					40.0	46.85		1.0		0.05	10.0	0.1	
88		1.1				40.0	47.80		1.00		0.05	10.0	0.1	
89		1.05				40.0	47.85	10.0	1.00	0.05	0.05			
90		1.05					98.95							
91	0.1	1.05					98.85							
92	0.3	1.05					98.65							
93	0.5	1.05					98.45							
94	1.0	0.50					98.50							
95	1.0	1.05					97.95							
96	1.0	2.10					96.90							
97		1.2				40.0	47.25	10.0	1.0	0.5	0.05			
98	2.0	0.3				40.0	46.55		1.0		0.05	10.0	0.1	
99	2.0	0.1				40.0	46.75		1.0		0.05	10.0	0.1	
100	2.0		1.2	1.9		40.0	43.79		1.0		0.01	10.0	0.1	
101			1.5			40.0	47.39		1.0		0.01	10.0	0.1	
102	2.0		4.0			40.0	42.89		1.0		0.01	10.0	0.1	
103			3.0			40.0	45.89		1.0		0.01	10.0	0.1	
104	2.0		3.0			40.0	43.89		1.0		0.01	10.0	0.1	
105			1.2			40.0	47.69		1.0		0.01	10.0	0.1	
106	2.0		0.2			40.0	46.69		1.0		0.01	10.0	0.1	
107	2.0		0.3			40.0	46.59		1.0		0.01	10.0	0.1	
108	2.0		0.5			40.0	46.39		1.0		0.01	10.0	0.1	
109							1.2	40.0	47.74	1.0	0.01	10.0	0.05	
110	2.0						0.5	40.0	46.44	1.0	0.01	10.0	0.05	

Table 2a - Combinations

	Herbicide no.	Carrier material	Additives	Mergal K9N
111	2.0	0.05	40.0	46.89
112	2.0	0.06	40.0	46.79
113	2.0	0.37	40.0	46.48
114		1.22	40.0	47.63
115		1.0	40.0	47.85
116		1.0	40.0	47.85
117		1.0	40.0	47.85
118	1.0	1.0	99.0	
119	2.0	1.0	98.0	
120	2.0	0.5	40.0	46.99
121		1.0	5.0	86.0
122	2.0	0.05	40.0	47.85
123	2.0	0.5	40.0	46.80
124		1.0	40.0	46.35
125	2.0	0.5	40.0	47.45
126	2.0	2.0	40.0	45.95
127	30.0		1.1	44.45
128			1.1	18.65
129			1.1	48.70
130			0.10	93.9
131			1.0	93.2
132			1.0	98.0
133			1.0	99.0
			40.0	47.85
			1.0	0.05
			10.0	10.0

Table 3a

	Herbicide	Carrier material	Additives
no.	Basta 50%	Aerogel P88	
134	2.0	40.0	46.35
135	2.0	40.0	44.85
136	2.0	40.0	46.75
137	2.0	40.0	45.85
138	2.0	40.0	46.44
139	2.0	40.0	46.25
140	2.0	40.0	46.84
141	2.0	40.0	46.65
			ammonium nitrate
			Bronidox L
			duasyn AE
			Geropon SDS
			Stepanol ME Dry
			Mergal K9N
			phenyl-succinic acid
			1-naphthyl-acetic acid Na
			3-indolyl-acetic acid Na
			gibberelic acid Na

B Biological examples

Meanings of the abbreviations used below:

5	ABUTH	<i>Abutilon theophrasti</i>	AMARE	<i>Amaranthus retroflexus</i>
	BRSNW	<i>Brassica napus</i>	CHEAL	<i>Chenopodium album</i>
	GALAP	<i>Galium aparine</i>	HORVS	<i>Hordeum vulgare</i>
	LOLMU	<i>Lolium multiflorum</i>		

10 **B.1 Action against harmful plants of glufosinate-ammonium bound to a polymer carrier material, compared to that of glufosinate-ammonium applied by a conventional method**

In a greenhouse, a granulous formulation, prepared according to example 48, of the herbicide glufosinate-ammonium was broadcast at an application rate of 500 g of active compound per hectare (converted) on humus-containing loam soil, and the soil was then watered. Three days after the application, seeds of *Setaria viridis* were sown at a depth of 1 cm. For the entire duration of the experiment, a daytime temperature of 22-24°C and a nighttime temperature of 16-18°C were maintained, a uniform day length of 16 hours being achieved by additional illumination with sodium vapor lamps (approximately 7000lux). The relative atmospheric humidity was 60-80%. Visual scoring 35 days after sowing showed that an activity of 70% against *Setaria viridis* was achieved. For comparison, glufosinate-ammonium was applied as an aqueous solution, under otherwise identical conditions and likewise at an application rate of 500 g of active compound per hectare (converted). Visual scoring 35 days after sowing showed that the activity achieved against *Setaria viridis* was nil (0%).

30 **B.2 Comparison of the action against harmful and useful plants of glufosinate-ammonium bound to polymer carrier material**

In a greenhouse, seeds of the harmful plants LOLMU, AMARE and CHEAL and the useful plant HORVS were sown on humus-containing loam soil, covered with a 0.5 cm layer of the same soil and then watered. One day after sowing, a formulation, prepared according to example 48, of the herbicide glufosinate-ammonium was broadcast on the soil at an application rate of 1000 g of active compound per hectare (converted). For the entire duration of the experiment, a daytime temperature of 22-24°C and a nighttime temperature of 16-18°C were maintained, a uniform day length of 16 hours being achieved by additional illumination with sodium

vapor lamps (approximately 7000 lux). The relative atmospheric humidity was 60-80%. Visual scoring 35 days after sowing gave the results shown in Table 5, and it was found that, in contrast to the three harmful plants tested, the crop plant HORVS had not been damaged by the herbicide.

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Table 5 Activity [%] against harmful and useful plants

Treatment with herbicide according to formulation example No.	LOLMU	AMARE	CHEAL	HORVS
48	40	70	60	0

B.3 Action against harmful plants of glufosinate-ammonium bound to different carrier materials

10 Seeds of the harmful plants GALAP, AMARE, CHEAL and LOLMU were sown outdoors on humus-containing loam soil, covered with a 0.5 cm layer of the same soil and then watered. One day after sowing, formulations, prepared according to examples No. 40, 45 and 46, of the herbicide glufosinate-ammonium were broadcast at an application rate of 750 g of
 15 active compound per hectare (converted) on the soil. Visual scoring 35 days after sowing gave the results shown in Table 6.

Table 6 Activity [%] against harmful plants

Treatment with herbicide according to formulation example No.	GALAP	AMARE	CHEAL	LOLMU
40	80	10	15	25
45	100	35	70	30
56	70	40	85	25

20 **B.4 Effect of different fertilizers on the efficacy of glufosinate-ammonium bound to polymer carrier material**

In a greenhouse, seeds of the harmful plants ABUTH, AMARE, CHEAL and LOLMU were sown in humus-containing loam soil, covered with a 0.5 cm layer of the same soil and then watered. One day after sowing, a formulation, prepared according to examples 41 and 43, of the herbicide glufosinate-ammonium was broadcast at an application rate of 750 g of active compound per hectare (converted) on the soil. For the entire duration of the experiment, a daytime temperature of 22-24°C and a nighttime temperature of 16-18°C were maintained, a uniform day length of
 25

16 hours being achieved by additional illumination with sodium vapor lamps (about 7000 lux). The relative atmospheric humidity was 60-80%. Visual scoring 35 days after sowing gave the results shown in Table 7.

5 Table 7 Activity [%] against harmful plants

Treatment with herbicide according to formulation example No.	ABUTH	AMARE	CHEAL	LOLMU
41	80	90	85	15
43	75	75	75	10

B.5 Effect of different surfactants on the efficacy of glufosinate-ammonium bound to carrier materials

In a greenhouse, seeds of the harmful plants GALAP, AMARE, CHEAL and 10 LOLMU were sown in humus-containing loam soil, covered with a 0.5 cm layer of the same soil and then watered. One day after sowing, a formulation, prepared according to examples 40, 29 and 28, of the herbicide glufosinate-ammonium was broadcast at an application rate of 15 750 g of active compound per hectare (converted) on the soil. For the entire duration of the experiment, a daytime temperature of 22-24°C and a nighttime temperature of 16-18°C were maintained, a uniform day length of 16 hours being achieved by additional illumination with sodium vapor lamps (about 7000 lux). The relative atmospheric humidity was 60-80%. Visual 20 scoring 35 days after sowing gave the results which showed that the efficacy of the herbicide is enhanced by adding surfactants to glufosinate-ammonium bound to carrier materials, see Table 8.

Table 8 Activity [%] against harmful plants

Treatment with herbicide according to formulation example No.	GALAP	AMARE	CHEAL	LOLMU
40 (without surfactant)	80	10	15	25
28 (with surfactant)	98	60	98	60
29 (with surfactant)	90	60	90	40

25 **B.6 Effect of preservatives on the efficacy of glufosinate-ammonium bound to polymer carrier material**

In a greenhouse, seeds of the harmful plants ABUTH and BRSNW were sown in humus-containing loam soil, covered with a 0.5 cm layer of the

same soil and then watered. One day after sowing, a formulation, prepared according to examples 40, 54 and 60, of the herbicide glufosinate-ammonium was broadcast at an application rate of 750 g of active compound per hectare (converted) on the soil. For the entire duration of the experiment, a daytime temperature of 22-24°C and a nighttime temperature of 16-18°C were maintained, a uniform day length of 16 hours being achieved by additional illumination with sodium vapor lamps (about 7000 lux). The relative atmospheric humidity was 60-80%. Visual scoring 35 days after sowing gave the results which showed that the efficacy of the herbicide is enhanced by adding preservatives to glufosinate-ammonium bound to carrier materials, see Table 9.

Table 9

Activity [%] against harmful plants

Treatment with herbicide according for formulation example No.	ABUTH	BRSNW
40 (without preservative)	30	0
54 (with preservative)	55	10
60 (with preservative)	55	70